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Minimum Ignition Temperature of Hybrid Mixtures

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Although the minimum ignition temperature is an important safety characteristic and hybrid mixtures are of high relevance in different industrial processes, the focus of research is directed towards single-substance systems. Unlike the situation in the industry the standards, too only focus on single-phase mixtures. To get minimum ignition temperatures for frequently used hybrid mixtures, first, the minimum ignition temperatures and ignition frequencies were determined in the Godbert-Greenwald furnace for two single-phase solids (corn starch and lycopodium) and a burnable gas (methane). Second, minimum ignition temperatures and ignition frequencies were determined for the combinations of the pure systems as a hybrid mixture of dust and gas.
By repeating each test point five times, the probability of ignition of the substance system could be analyzed in addition to the Minimum Ignition Temperature. No noticeable decrease of minimum ignition temperatures below the MIT of the pure solids was observed for the hybrid mixture consisting of methane and starch or lycopodium respectively, but a more widely dispersed area of ignition is shown. In accordance with previous findings, the results demonstrate a strong relationship between the likelihood of explosion and the amount of added gas. In consequence, the hybrid mixture is characterized by a minimum ignition temperature that is dominated by the lower igniting component of the mixture.

1. Introduction

The impact of hybrid mixtures on safety characteristics is important for several industrial processes, e.g., in mining (Li et al., 2012), pharmaceutical production (Hossain et al., 2014) or metal processing (Nifuku et al., 2007). They consist of a component of a combustible dust and a flammable gas and may form an explosive mixture below the minimum explosible concentration or lower the flammability limit respectively (Amyotte and Eckhoff, 2010). As the presence of hot surfaces in combination with ignitable dust clouds is frequently relevant in industrial processes (e.g. sawing, cutting, grinding, drying or heating of particles), the Minimum Ignition Temperature (MIT) is an important property because the combustion reaction of the substances becomes self-sustaining only above this temperature (Amyotte, 2013). Bartknecht (1981) emphasized that a two-component mixture of a flammable dust and gas can be ignited as a mixture below the lower explosion limit of every single component. Zunaid et al. have been presented a substantial amount of spark formation below the MIT as a potential ignition source for hybrid mixtures (Zunaid, 2013). Effects of ignitable dust-vapor-mixtures to explosions with greater severity than the ones of both compounds taken separately were presented by Dufaud et al. (Dufaud et al., 2008; 2009). A substantial increase of knowledge about the minimum ignition temperature of different hybrid mixtures were presented by Addai et al. (2016a; 2016b; 2017).
Findings of minimum ignition temperature for premixed dust-solvent mixtures were presented by Gabel and Krause (2019). Gabel et al. (2021) demonstrate a strong relationship between the likelihood of explosion and the amount of added solvent. They found a noticeable decrease of minimum ignition temperatures below the MIT of the pure solids for a hybrid mixture consisting of corn starch and n-heptane.

Little is known about whether
1) the classical GG furnace is suitable to generate MITs for hybrid mixture systems and
2) what number of replicate experiments is required to obtain robust results.

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In the presented study MIT was determined for three pure systems: dehumidified corn starch and Lycopodium as dust particle samples and methane as a gas system. Given the MIT of pure dust particle systems, the MIT based on a variated admixture of methane was determined.

2. Materials and apparatus

Corn starch (Figure1, left) was chosen in the NexHys project as standard, and the same sample is used by all partners for all experiments in the project. Lycopodium (Figure 1, right) on the other hand serves as common dust in dust explosion testing for decades. Even if the latter has no industrial relevance, it assures certain comparability to existing publications. Methane was used as a gas component as it is of high relevance in agriculture, power generation, and chemical process industry.

2.1 Materials

Studies with five repetitions of each single data point were performed for pure dust systems of dehumidified corn starch and Lycopodium and a hybrid mixture of dust combined with methane.

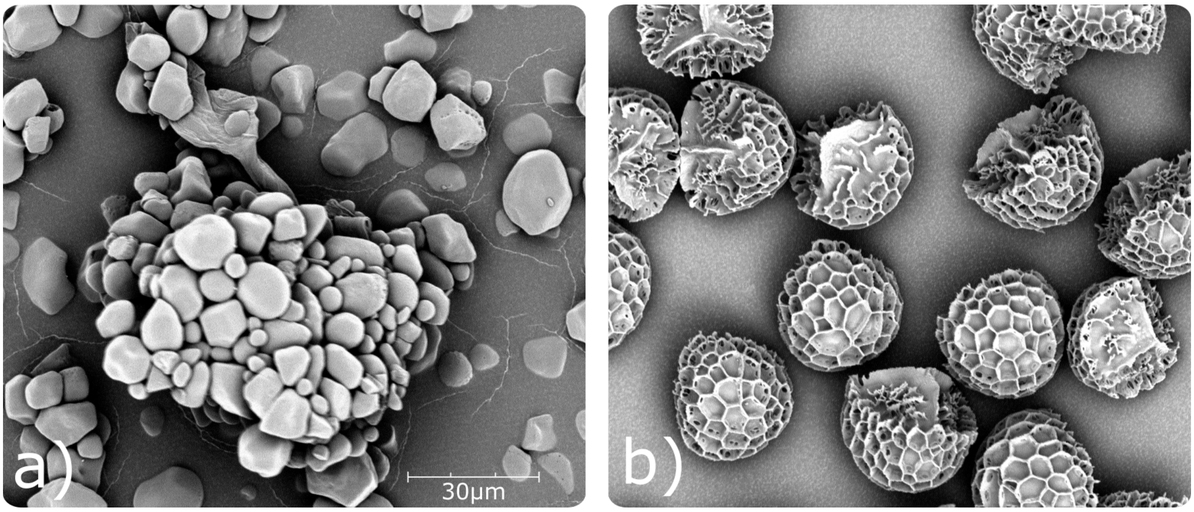

Figure 1: Images captured at 2000x magnification with a scanning electron microscope of (a) NexHysStarch and (b) Lycopodium.

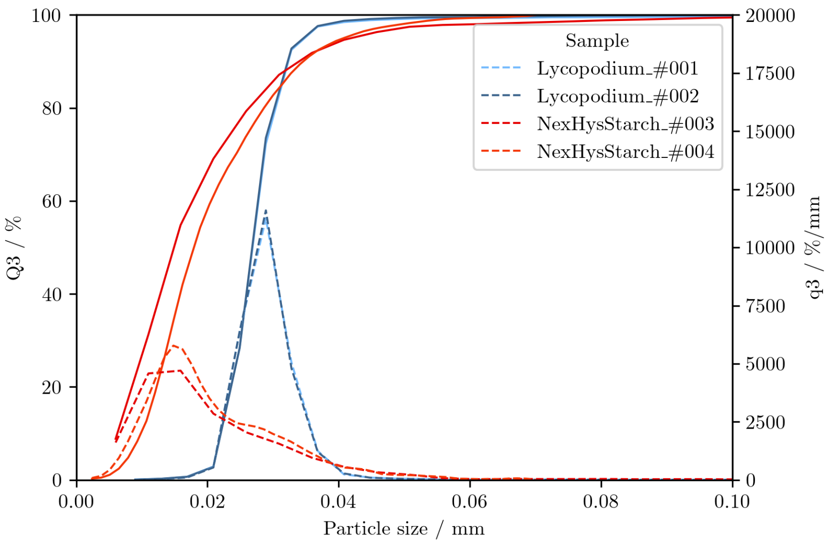

Figure 2: Particle size distribution of NexHys corn starch (red) and Lycopodium (blue), two measurements each.

The particle size distribution for the dust samples were determined according to ISO 13322-2:2006-11, 2006 (cf. Figure 2). The dust sample was characterized by a median particle size of approximately 0,017 mm (NexHys corn starch) and 0,031 mm (Lycopodium). The residual humidity was determined by Satorius MA 100 for the dust samples. Median residual moisture of approximately 7.74 % after two hours for NexHys corn starch and 3.16 % for Lycopodium was measured.

2.2 Experimental setup and procedure

All MIT tests were conducted in a modified GG oven (Figure 3) by following DIN 50281-2-1, 1999 (Methods for determining the minimum ignition temperatures of dust. Method B: Dust cloud in a furnace at a constant temperature). The Godbert-Greenwald furnace is 216 mm high, measures 36.5 mm in internal diameter, and consists of a silica glass tube that can be heated from the outside. Inside the tube are two thermocouples for control and temperature measurement. The furnace opens downwards to the atmosphere and is connected to the dust storage vessel by a 90° angled glass elbow. The distributing air blast is generated by pressurized air from a 500 cm^3 vessel separated by a solenoid valve. To detect an ignition the flames can be seen upon leaving the tube or via the mirror below the opening.

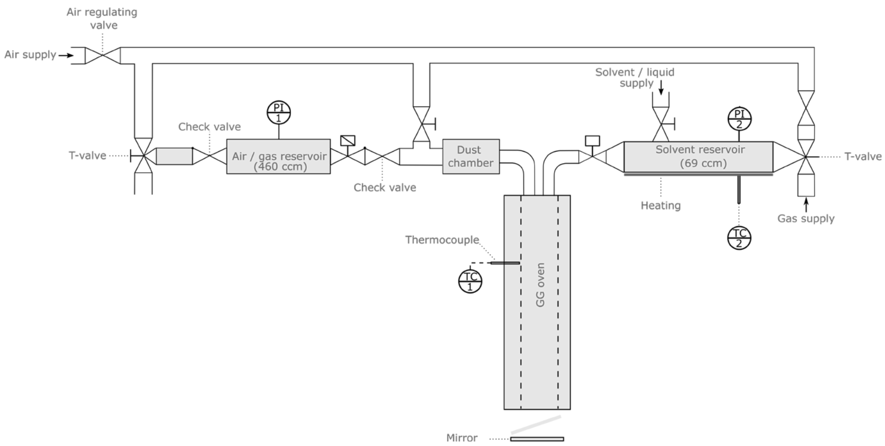

Figure 3: Schematic drawing of the modified Godbert-Greenwald-apparatus

For pure gas experiments and hybrid mixtures, the necessary gas concentration was generated in the Solvent reservoir by the partial pressure method. All gas concentrations stated in this paper revere only to the concentration in this vessel. The concentration during the ignition experiment in the tube is unknown.
The same holds true for the dust concentration. As the authors deny a homogeneous distribution of the dust in the tube no dust concentrations are specified. Thus, only the weight of dust in the Dust chamber is given.
Furthermore, it must be stated that the amount of dust chosen for the experiment is not equal to the amount of dust distributed in the tube as considerable residues can be found in the Dust chamber after each experiment. The Dust chamber used in these experiments is modified and optimized to ensure lower remains.
For experiments without dust, the same overpressure as in the Solvent chamber is set in the air reservoir.

3. Results

3.1 Pure substances

First, the MIT of the pure dust was determined. Unlike the standard procedure, more repetitions per measurement point were made to be able to account for the ignition ratios. These are visible in the diagrams by the size and the color of the scatter points. Additionally, the Frequencies in x and y direction are shown in the histogram plots at the margins of the diagrams.
The accumulated ignition ratio counts of pure corn starch (Figure 4, left) and Lycopodium (Figure 4, right) depending on the mass and the temperature of the GG furnace is presented. As expected, the ratio of ignitions decreases depending on temperature, and the probability to ignite increases with an increasing amount of dust. It can be observed that there is a dust concentration level at which the ignitability cannot be increased by further increasing the dust mass (c_{Starch} = 0.5 g, $c_{Lycopodium}$ = 0.4 g). Theoretically, one would expect a decrease in the likelihood of ignitions if the amount of dust increases. This expectation could not be confirmed in the experiments presented here, as the injection pressure would need to be higher and the remaining dust amount in the chamber is increasing differently in practice.

A well-defined non-ignition temperature is observed for corn starch at 380°C and Lycopodium at 420°C respectively. These limits are confirmed by 27 repetitions without any ignitions.

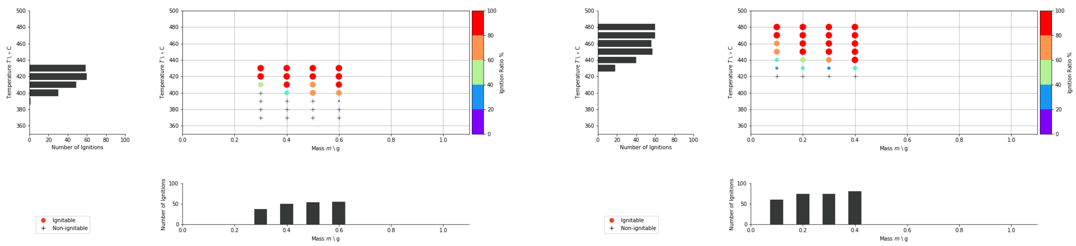

Figure 4: Ignition ratio counts of pure corn starch depending on mass and temperature of the GG furnace (left). Please note that the number of ignitions can be greater than five, as the data has been grouped by the varied injection pressures (p^+ = [0:3; 0:5; 0.7] bar). Ignition ratio counts of pure Lycopodium depending on mass and temperature of the GG furnace (right). Please note that the number of ignitions can be greater than five, as the data has been grouped by the varied injection pressures (p+ = [0:3; 0:5; 0.7] bar).

The MIT of Methane is measured to be 660°C in this experimental setup (Fig. 5) and recognizable above the standard value of 595°C (Gestis).

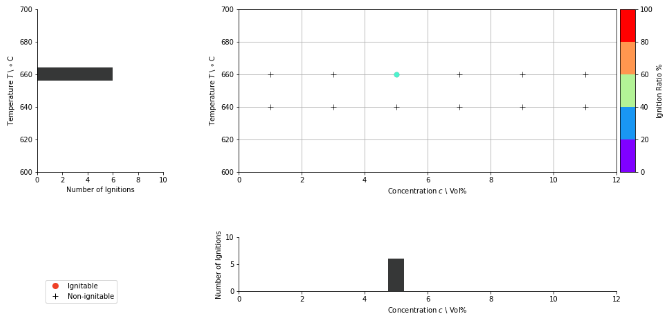

Figure 5: Ignition ratio counts of pure Methane depending on concentration and temperature of the GG furnace. Frequencies in the x and y direction are shown in the histogram plots at the margins.

As presented in Figure 5, the ideal concentration for ignition is 5 %, which is half of the stoichiometric concentration. As a higher dilution can be assumed in the experiment, it can be assumed that the effective gas concentration in the combustion chamber now of ignition is even lower. The determining influence of the high temperature on the combustion process is highlighted here.

3.2 Hybrid mixtures

Hybrid mixtures of Methane combined with corn starch and or Lycopodium were tested. Due to the higher MIT of the gas, an increase in MIT was not expected for the hybrid mixture system. The focus was on whether the amounts of dust required to achieve an ignition would change compared to the pure dust experiments. To also limit the parameter space to be tested for research pragmatic concerns, the injection pressure p+ was set as constant by 0.5 bar. This is in line with a series of preliminary tests (Ernst (2020), Hofmann (2020)) and following the findings of the experiments with pure dust systems. As part of the research project, the entire parameter range was systematically tested (publication of the results is planned for 2022). The slight increase of the MIT detected for both systems might be due to the reduced level of oxygen in the system, but still lies within the measurement uncertainty.
Regarding the systems from the side of the gas, it can be shown, that even very low gas concentrations lead to an ignition where the pure gas system needs higher concentration to allow self-sustaining flame propagation.

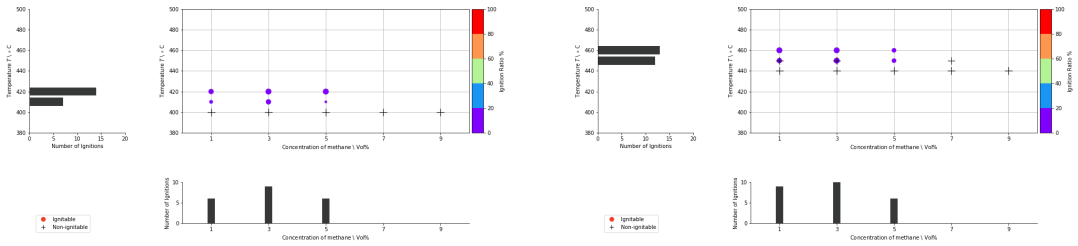

Figure 6: Cumulative ignition counts of a hybrid mixture consisting of m = [0.4, 0.5] g corn starch and a variable concentration of methane in a temperature interval of T = [400; 420] °C (left). Cumulative ignition counts of a hybrid mixture consisting of m = [0.2, 0.4] g Lycopodium and a variable concentration of methane in a temperature interval of T = [440; 460] °C (right).

A comparison of the MIT of hybrid mixtures is shown in Fig. 6. A slight increase in ignition temperatures with an increasing gas concentration in the hybrid mixture is observed compared to the pure substances.

4. Conclusions and Outlook

The present experimental setup is suitable to test for the MIT of hybrid mixtures. The changes applied do not influence the standard procedure for pure dust and enable users to test for hybrid MIT of dust and gases (as well as for vapors, even if not presented here). The necessary number of non-ignitions to be given in a revised standard still needs to be discussed and is a function of the step width of the parameters of the experiment.
Systematic measurements of the MIT in the GG oven are very time-consuming if additional points and more repetition are realized to make results more reliable. One aim of the NexHys is to generate a scientific basis to justify a final low number of repetitions for the standard procedure.
The MIT of the hybrid system is located close to the component with the lower ignition temperature (in our case: the dust components). An influence of the gas concentration on the ignition probability could not be shown in the experiments: even a small concentration of methane favored ignition (which, however, is still higher than the MIT of the pure dusts). Additionally, a wider region of transition between ignition and non-ignition can be achieved. In consequence, a hybrid mixture system consisting of gas and solid is characterized by a different minimum ignition temperature than that of the single components.
Further combination with gases or vapors ignitable at lower temperatures than the dust will follow. This should prove the expected general behavior postulated before. Most of all no synergistic effect should to be found, with a hybrid MIT below the MITs of the single components. Therefore, the standardization committees will be involved and ask for input, concerning possible critical substance combinations as well as further mixtures of interest.

Nomenclature

GG – Godbert-Greenwald Furnace
MIT – Minimum Ignition Temperature, °C
p^+ - injection pressure, bar

Acknowledgments

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